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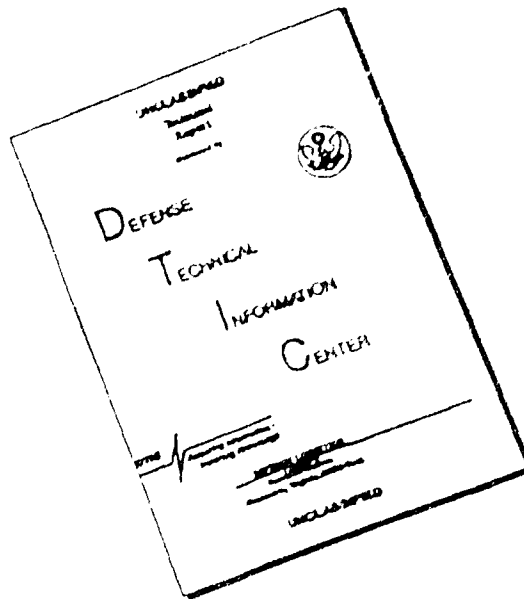
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Final Report

on

# **Alloys for High-Temperature Service**

by

**R. L. Beck, E. E. Fletcher, A. R. Elsea  
A. B. Westerman and G. K. Manning  
Battelle Memorial Institute**

**In Cooperation With  
Office of Naval Research, Navy Department  
Contract No. N5ori-111  
April 30, 1952**

FINAL REPORT

on

ALLOYS FOR  
HIGH-TEMPERATURE SERVICE

to

OFFICE OF NAVAL RESEARCH,  
NAVY DEPARTMENT

April 30, 1952

Contract No. N5ori-111, Task Order I  
Project NR 031-003

by

R. L. Beck, E. E. Fletcher, A. R. Elsea,  
A. B. Westerman, and G. K. Manning

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## TABLE OF CONTENTS

	<u>Page</u>
INTRODUCTION . . . . .	1
SUMMARY . . . . .	2
EXPERIMENTAL PROCEDURE AND RESULTS . . . . .	4
Study of Phase Relationships in Cobalt-Chromium-	
Base Binary and Ternary Alloys . . . . .	4
Study of Phase Relationships in Vitallium (Stellite No. 21) . .	17
Mechanical Properties of Cobalt-Chromium-Base Alloys . .	19
Creep-Rupture Tests on Binary and Ternary Alloys . .	19
Properties of Stellite No. 21-Type Alloys . . . . .	21
Room-Temperature Hardness . . . . .	21
Creep-Rupture Data . . . . .	22
Hot-Hardness Data . . . . .	22
APPENDIX	

FINAL REPORT

on

ALLOYS FOR HIGH-TEMPERATURE SERVICE

to

OFFICE OF NAVAL RESEARCH, NAVY DEPARTMENT  
Contract No. N5ori-111, Task Order I  
Project NR 031-003

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BATTELLE MEMORIAL INSTITUTE

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R. L. Beck, E. E. Fletcher, A. R. Elsea,  
A. B. Westerman, and G. K. Manning

April 30, 1952

INTRODUCTION

A research investigation was undertaken at Battelle Memorial Institute, under the sponsorship of the Office of Naval Research, to study the fundamental factors promoting high-temperature strength in alloys. The alloys of this type in general use today have been developed on an empirical basis, without a clear understanding of the structural factors responsible for their high-temperature properties. The best high-temperature alloys now in use contain considerable quantities of cobalt, nickel, and chromium, all of which are of strategic importance. If the basic factors responsible for high-temperature strength were understood better, undoubtedly less strategic alloys with better high-temperature properties could be designed.

At the time this investigation was started, the cobalt-chromium-base alloy, Vitallium, was one of the best high-temperature alloys available. Consequently, this investigation was to comprise a study of the relationship between the structure and properties of this alloy. Since Vitallium is such a complex material, it was necessary to obtain information regarding the phase relationships in the pertinent binary and ternary systems before starting a structural analysis of this alloy.

The reports issued and the articles published on the results of this research are listed in Appendix I.

## SUMMARY

A metallographic and X-ray diffraction study was made to determine the cobalt-chromium binary diagram. The effects of ternary additions of nitrogen, iron, nickel, molybdenum, or tungsten, all components of Vitallium-type alloys, on the reactions which occur in the cobalt-rich terminal solid solutions of this binary system were established also.

It was found that the transformation and precipitation reactions in these alloys were extremely sluggish. The addition of chromium raised the temperature of the alpha-beta transformation in cobalt. When nitrogen, iron, or nickel was added to cobalt-chromium alloys, the alpha-beta transformation was lowered markedly. Additions of tungsten had little effect on the transformation, while molybdenum raised the transformation temperature.

The cobalt-chromium intermetallic compound, gamma ( $\text{Co}_2\text{Cr}_3$ ), precipitated from the beta phase, the low-temperature modification of the terminal solid solution, at very low chromium concentrations. This gamma precipitate sometimes occurred as small spheroids, while in other cases it took the form of platelets which were components of a lamellar structure resembling pearlite. In general, all of the ternary additions studied tended to raise the temperature at which gamma started to precipitate from solid solution.

Although nitrogen is not added to Vitallium-type alloys intentionally, its effect was studied, because early in this investigation it was found that considerable nitrogen is picked up by these alloys when processed in air. Since nitrogen lowers the alpha-beta transformation temperature range and alters the temperature at which gamma precipitation occurs, variations in nitrogen content may account for the erratic properties reported for these alloys.

Metallographic and X-ray diffraction studies were conducted to determine the phase relationships in Stellite No. 21 (Vitallium) after various heat treatments. A method of heat tinting these alloys for metallographic identification of the minor phases was developed. These minor phases included gamma phase ( $\text{Co}_2\text{Cr}_3$ ) and three carbides ( $\text{M}_{23}\text{C}_6$ ,  $\text{M}_6\text{C}$ , and  $\text{Cr}_7\text{C}_3$ ). The upper limit of the alpha-beta transformation temperature range in the Stellite No. 21-type alloys was found to be about  $1000^\circ\text{C}$ . Nickel additions had considerable effect in lowering the upper limit of this range, while carbon and molybdenum were quite effective in raising it. Only the upper limit of the transformation range could be determined because of the sluggish nature of this reaction at low temperatures. However, there were indications that the lower limit was approximately  $700^\circ\text{C}$ . The  $\text{Cr}_7\text{C}_3$ -type carbide was found in the as-cast alloy and in both the as-cast and homogenized material after aging at relatively low temperatures. The  $\text{M}_6\text{C}$ -type carbide was found in all the specimens aged at lower temperatures; however, it was taken into

solution at aging temperatures above 1150°C. The  $M_{23}C_6$ -type carbide remained out of solution at considerably higher temperatures.

Creep-rupture tests were made on cobalt-chromium alloys with and without nitrogen at 780, 915, and 1000°C., and on Stellite No. 21-type alloys at 816°C. Increasing the chromium, carbon, or nitrogen content of these alloys increased their creep resistance at all temperatures tested. The Stellite No. 21-type alloys were stronger in the as-cast condition than in any homogenized or homogenized and aged condition tested. Suitable aging treatments prior to testing further increased the high-temperature strength of the as-cast material.

The results of these tests indicated that alpha and beta phases had about the same strength at elevated temperatures. However, a mixed alpha-beta structure was very weak. It is interesting to note that the normal operating temperatures for gas-turbine blades are in the range where these weak mixed structures are found in Stellite No. 21 alloy.

Some of these alloys were found to have lower creep rates at high stresses than at lower stresses in tests conducted at the same temperatures. Metallographic examination of the specimens after test showed that the precipitation of gamma phase was affected by the stress. At higher stresses the gamma particles were smaller and more numerous, while at lower stresses there were relatively few large gamma particles. This difference in size of the precipitated gamma phase probably influenced the creep rate.

The results of other creep-rupture tests showed that the degree of prestress at the testing temperature influenced the creep rate of these alloys. The higher the prestress, the lower was the creep rate at a particular temperature. This occurred even at temperatures above those at which gamma precipitate formed. Consequently, some factor other than the precipitation of gamma was responsible for this behavior.

First-stage creep seldom was observed in the time-deformation curves for the binary and ternary alloys. There was usually a dip in the curve prior to the onset of second-stage creep and, in some instances, actual contraction of the test specimen occurred. An analysis of these data showed that the time-deformation curves for these materials probably represented the summation of two opposing reactions—one, a normal tendency toward creep, including the usual first-stage creep, and, second, a tendency toward contraction which may result from the precipitation of submicroscopic particles.

The influences of composition and prior treatment upon the aging characteristics of Stellite No. 21-type alloys were determined. It was found that minor changes in carbon, nickel, or molybdenum had little effect on the aging characteristics of these alloys.

## EXPERIMENTAL PROCEDURE AND RESULTS

### Study of Phase Relationships in Cobalt-Chromium-Base Binary and Ternary Alloys

A survey of the literature showed that the available published diagrams were unreliable. Consequently, the first phase of this fundamental study consisted of determining the cobalt-chromium binary diagram.

Metallographic and X-ray diffraction studies were made to determine the cobalt-chromium binary system and the effects of ternary additions of nitrogen, iron, nickel, molybdenum, or tungsten on the reactions which occur in the cobalt-rich terminal solid solutions. The materials used for this study were prepared from the purest melting stock available. Small laboratory melts were made either in a vacuum-induction unit or under a controlled atmosphere. The resulting alloys were cast either as small ingots, which were forged into small bars, or directly into bars. This bar stock was subjected to a homogenizing treatment to eliminate segregation. Further details regarding the analysis of the melting stock, the melting and casting procedures, the composition of the alloys made, and the homogenizing procedure are given in the Summary Reports dated November, 1947, September, 1949, and December, 1950.

Small specimens of the homogenized bar stock were subjected to aging treatments at various temperatures for times ranging from 50 to 100 hours. Metallographic examination of these specimens was made to determine the phases present\*. It was found that the reactions in this system were very sluggish. Consequently, experiments were performed to determine the time required to reach equilibrium in one of the alloys at a temperature in the alpha-beta transformation range. It was found that approximately 31 days would be required to reach equilibrium, and that the rate of transformation from alpha to beta was about equal to the rate of transformation from beta to alpha\*\*. To avoid these long aging times, the factor of sluggishness was overcome by approaching equilibrium from two directions, i.e., from higher and from lower temperatures\*\*\*.

In these alloys, the observed phases which are pertinent to Vitallium were as follows: Alpha phase is the high-temperature modification of the cobalt-rich terminal solid solution. It has a face-centered cubic lattice and

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\* The metallographic etchants used to identify the phases encountered in this system are described in the Summary Report dated November, 1947, on pages 11 to 18.

\*\* These experiments are described in the Summary Report dated November, 1947, on pages 18 through 23.

\*\*\* The method used is presented in detail in the Summary Report dated November, 1947, on pages 23 through 25.

undergoes a martensite-type transformation to beta phase upon cooling. Beta phase is the low-temperature modification of the cobalt-rich terminal solid solution. It has a hexagonal-close-packed lattice. Gamma phase is a brittle intermetallic compound of cobalt and chromium corresponding approximately to the ratio  $\text{Co}_2\text{Cr}_3$ . It has a complex crystal structure, the actual nature of which was not determined. Gamma is isomorphous with the sigma phase found in the iron-chromium system. Two other phases were found in the binary system which were not pertinent to Vitallium. These are epsilon and delta\*.

The results of the structural analysis of the heat-treated alloy specimens were used to plot the cobalt-chromium binary diagram shown in Figure 1, and portions of the ternary diagrams for cobalt-chromium-base alloys containing nitrogen, iron, nickel, molybdenum, or tungsten, shown in Figures 2 through 11.

Figure 1 indicates that the addition of chromium to cobalt raised the alpha-beta transformation temperature range. Over the composition range of interest to the study of Vitallium-type alloys, the transformation varied from approximately 800 to 925°C., depending upon the chromium content. It also is shown that the temperature range over which beta existed as a single phase free from gamma was very narrow; over the composition range of interest to Vitallium-type alloys this temperature range was a band 25 to 52°C. wide. Below this temperature range, gamma precipitated either as small spheroidal particles or as platelets; in the latter case, a lamellar structure was formed which resembled pearlite.

Figures 2 through 7 show that additions of nitrogen, iron, or nickel lowered the alpha-beta transformation temperature range for alloys with  $\frac{\text{Cr} \times 100}{\text{Co} + \text{Cr}} = 32$  and 20 per cent, and Figures 8 and 9, that tungsten had relatively little effect on the alpha-beta transformation temperature range. However, the addition of approximately 6 per cent tungsten to these alloys raised the temperature at which gamma started to precipitate by about 90°C. Figures 10 and 11 indicate that additions of molybdenum raised the alpha-beta transformation temperature range. In this respect, the effect of molybdenum was similar to that of chromium. The addition of molybdenum also raised the temperature at which gamma phase started to precipitate in the alloys at both chromium levels. However, this effect was much more pronounced in the higher chromium alloy.

Further studies of ternary and quaternary systems leading toward the synthesis of Vitallium-type alloys would undoubtedly have become quite complex. Furthermore, when attempts were made to calculate the alpha-beta transformation temperature in some of the more complex alloys using data obtained from the investigation of the binary and ternary systems, some discrepancies were encountered which indicated that there may be interactions between some of the various components. Consequently, it was decided that

\* These are described in the Summary Report dated November, 1947, on pages 14 through 16.



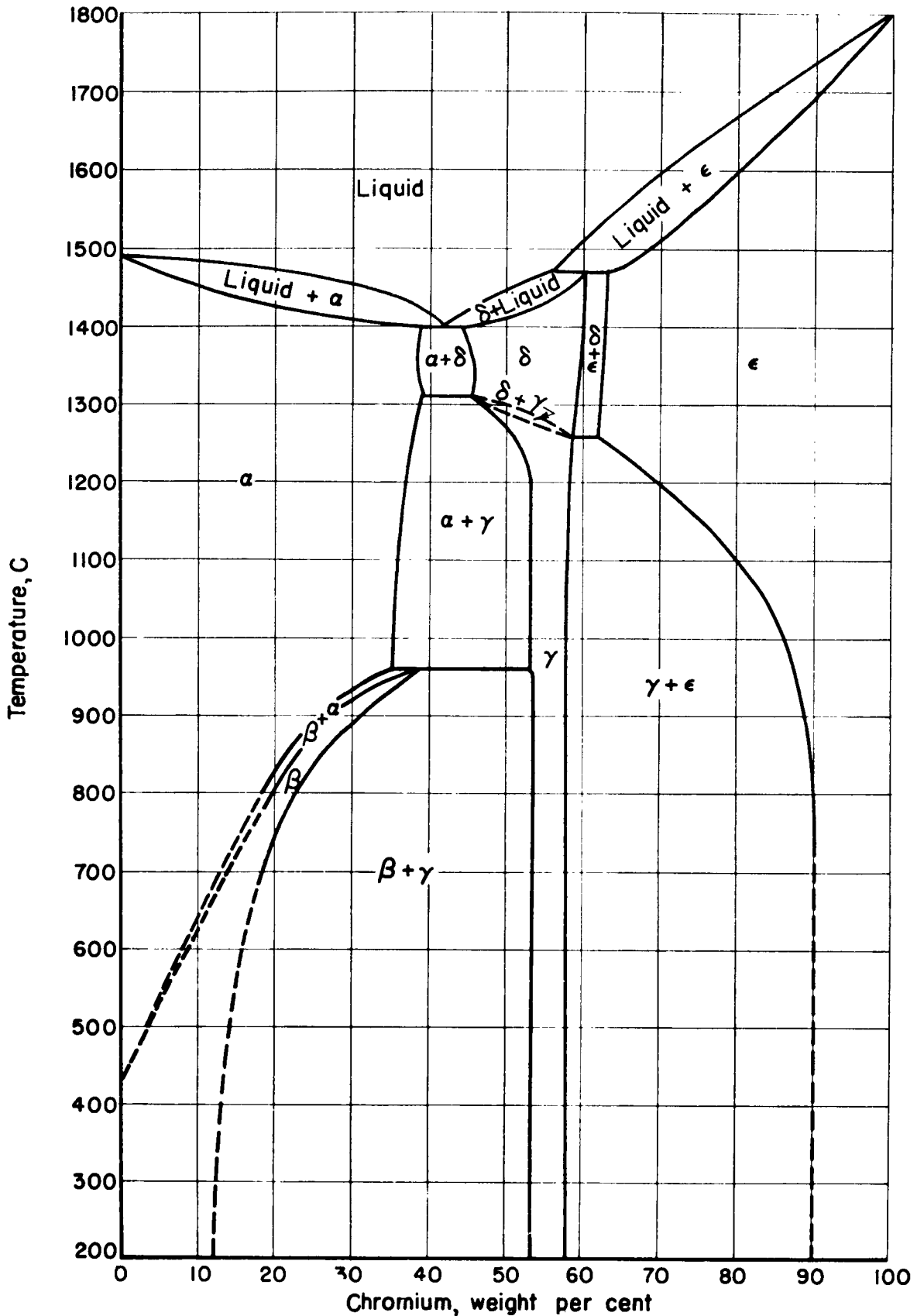


FIGURE 1. EQUILIBRIUM DIAGRAM FOR THE COBALT-CHROMIUM BINARY SYSTEM

A-1938

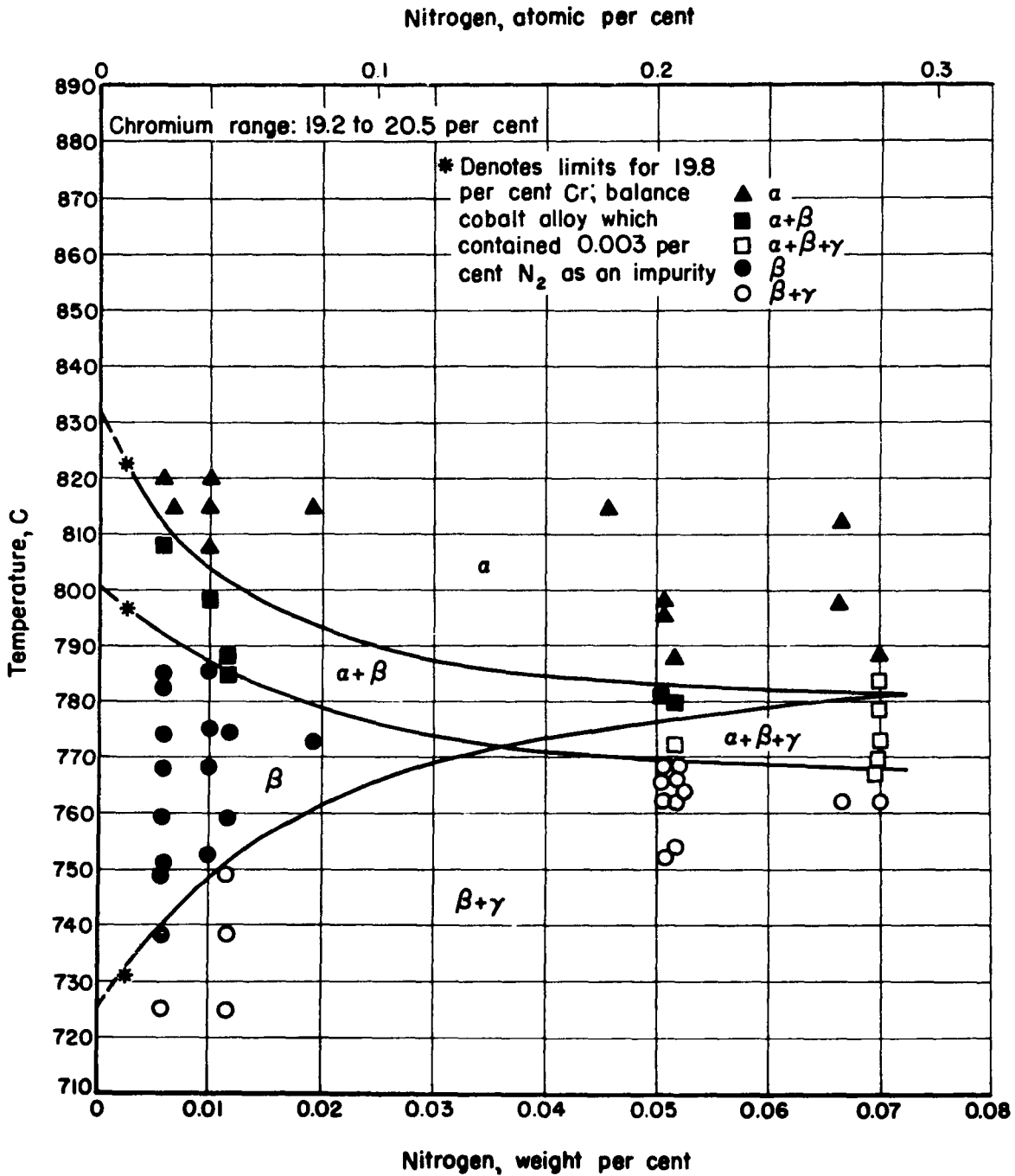


FIGURE 2. ALPHA-PLUS-BETA TEMPERATURE RANGE AND GAMMA PRECIPITATION AS A FUNCTION OF NITROGEN CONTENT FOR COBALT-CHROMIUM-NITROGEN ALLOYS CONTAINING 19.2 TO 20.5 PER CENT CHROMIUM

A-1939

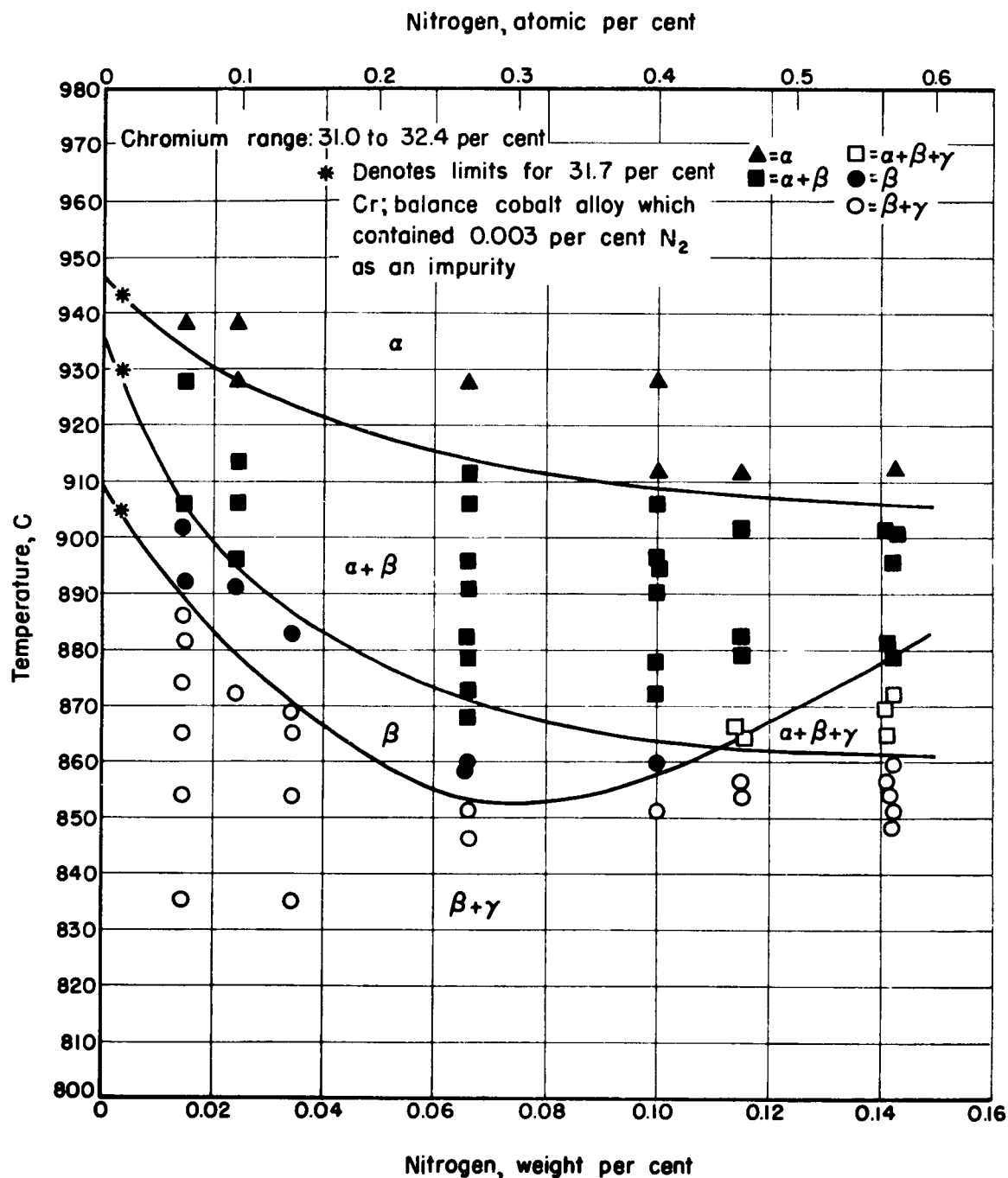


FIGURE 3. ALPHA-PLUS-BETA TEMPERATURE RANGE AND GAMMA PRECIPITATION AS A FUNCTION OF NITROGEN CONTENT FOR COBALT-CHROMIUM-NITROGEN ALLOYS CONTAINING 31.0 TO 32.4 PER CENT CHROMIUM

A-1940

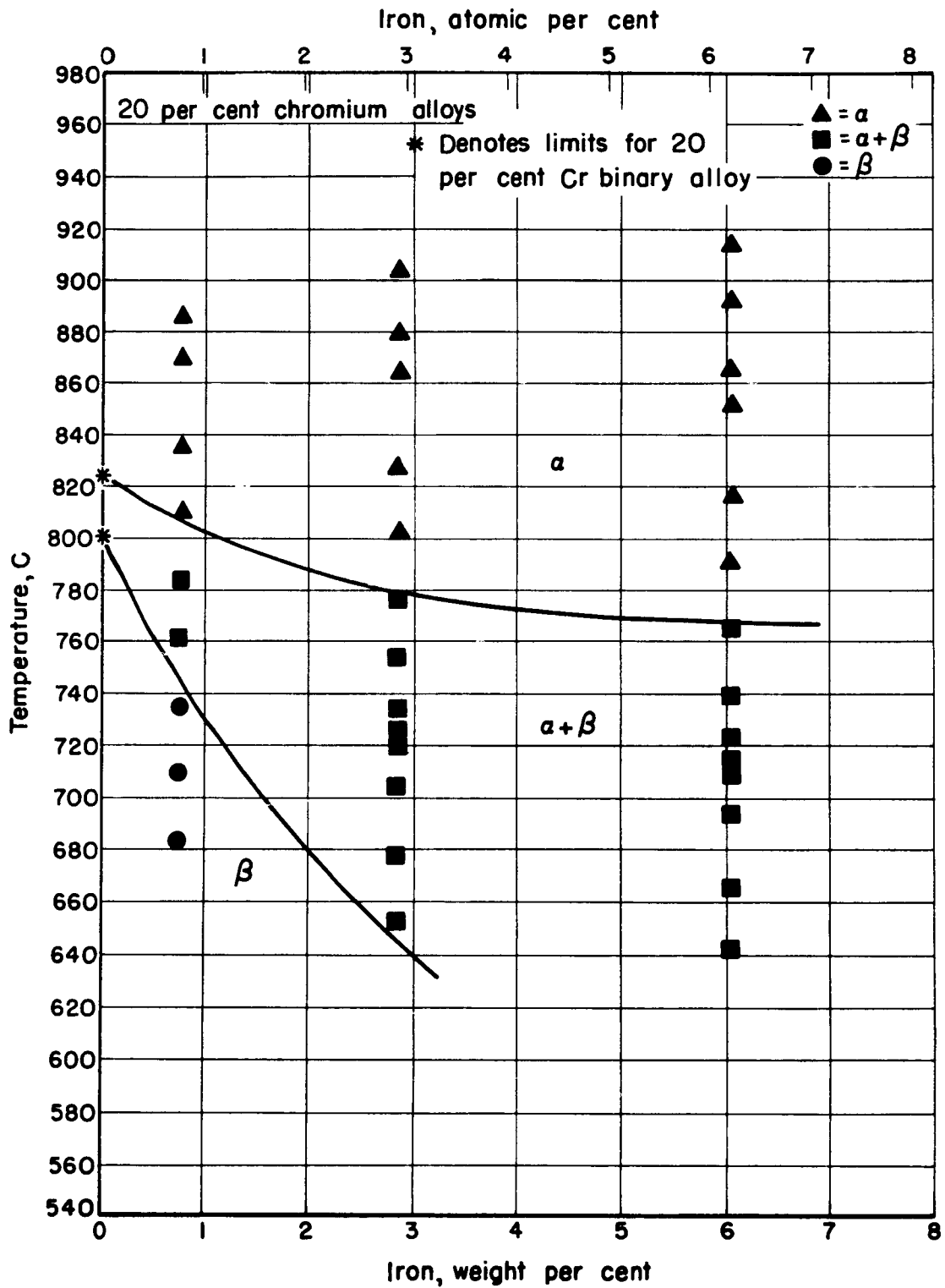


FIGURE 4. ALPHA-PLUS-BETA TEMPERATURE RANGE AS A FUNCTION OF IRON CONTENT FOR COBALT-CHROMIUM-IRON ALLOYS CONTAINING 20 PER CENT CHROMIUM

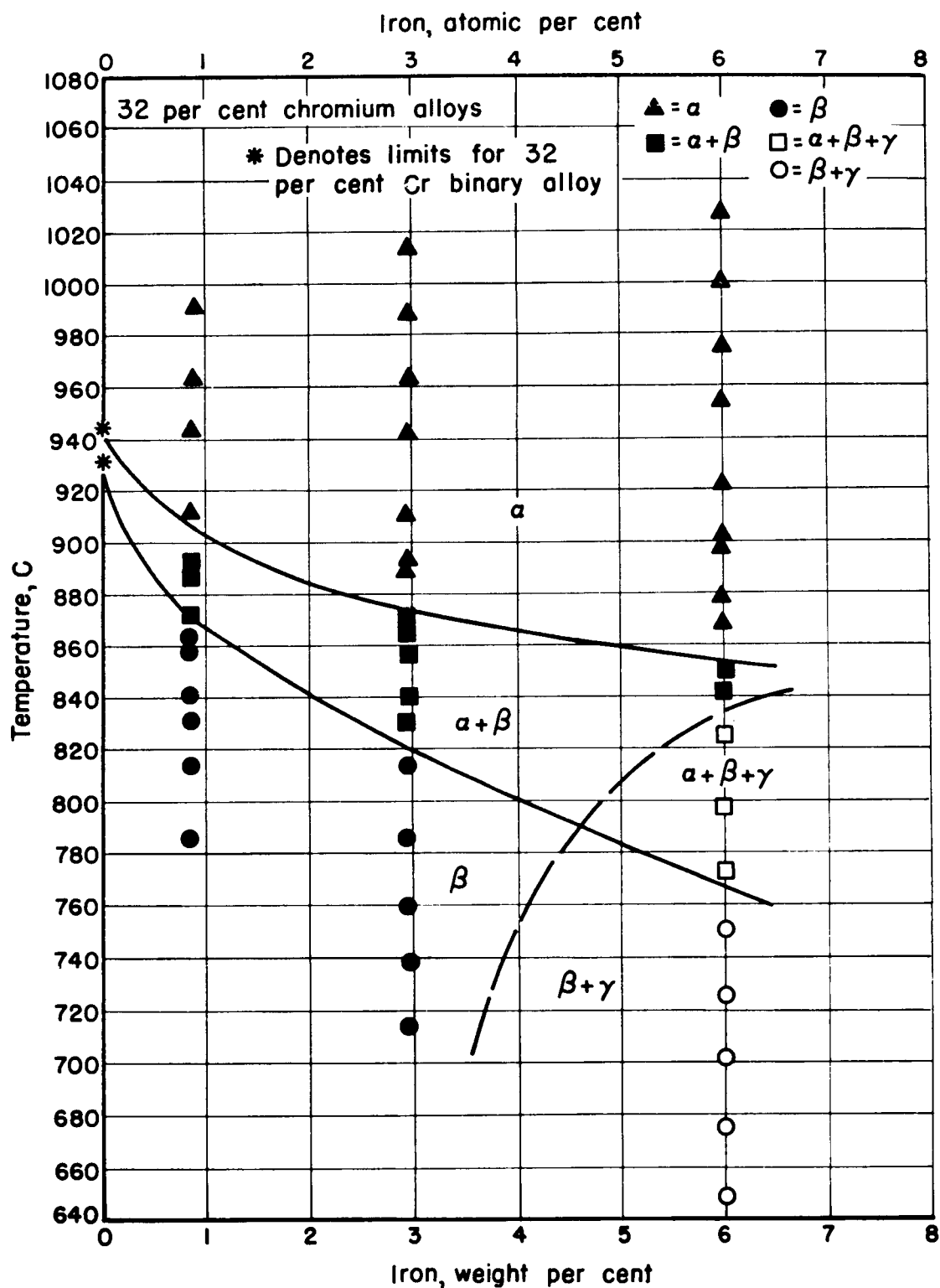


FIGURE 5. ALPHA-PLUS-BETA TEMPERATURE RANGE AS A FUNCTION OF IRON CONTENT FOR COBALT-CHROMIUM-IRON ALLOYS CONTAINING 32 PER CENT CHROMIUM

A-1942

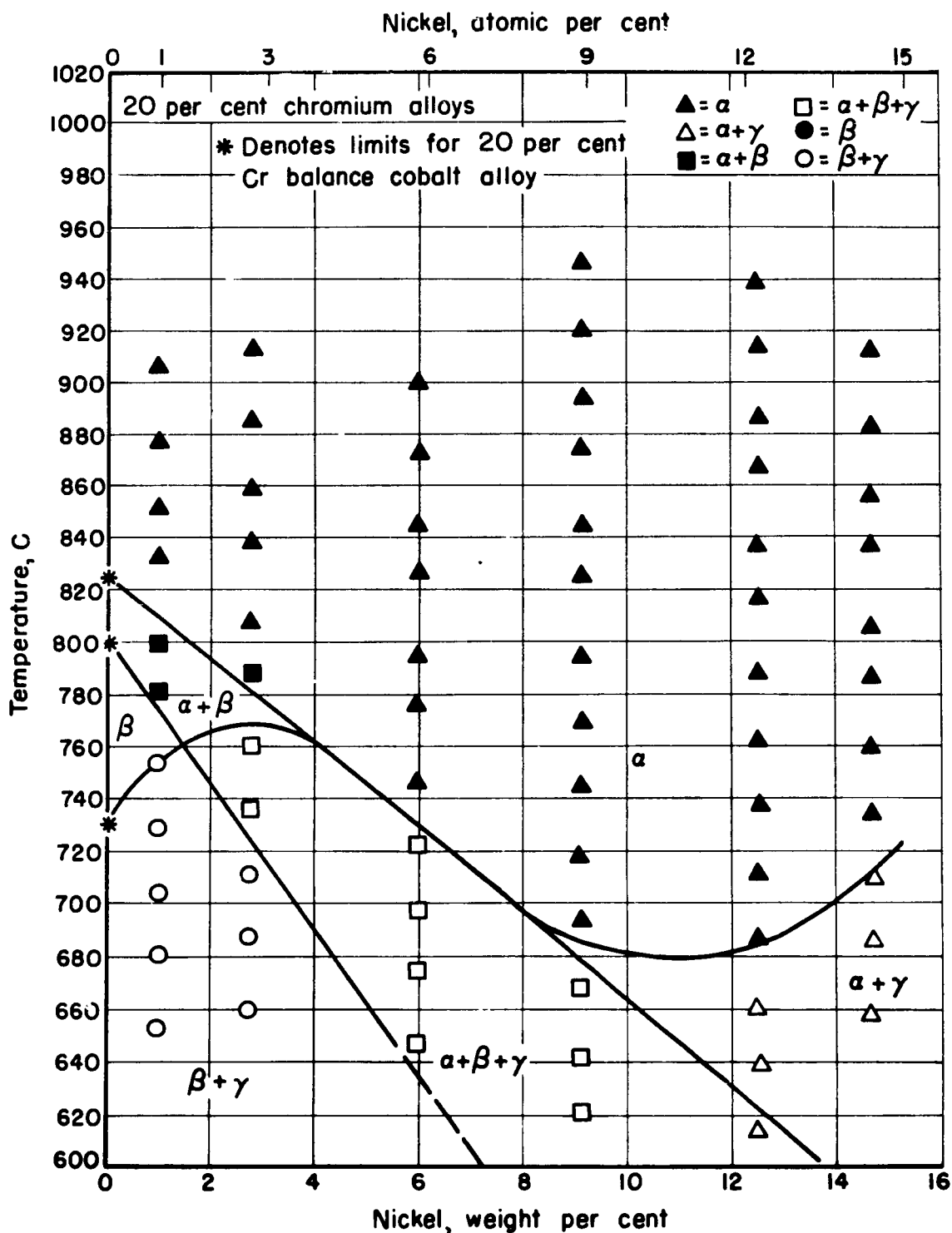


FIGURE 6. ALPHA-PLUS-BETA TEMPERATURE RANGE AND GAMMA PRECIPITATION AS A FUNCTION OF NICKEL CONTENT FOR COBALT-CHROMIUM-NICKEL ALLOYS CONTAINING 20 PER CENT CHROMIUM

A-1943

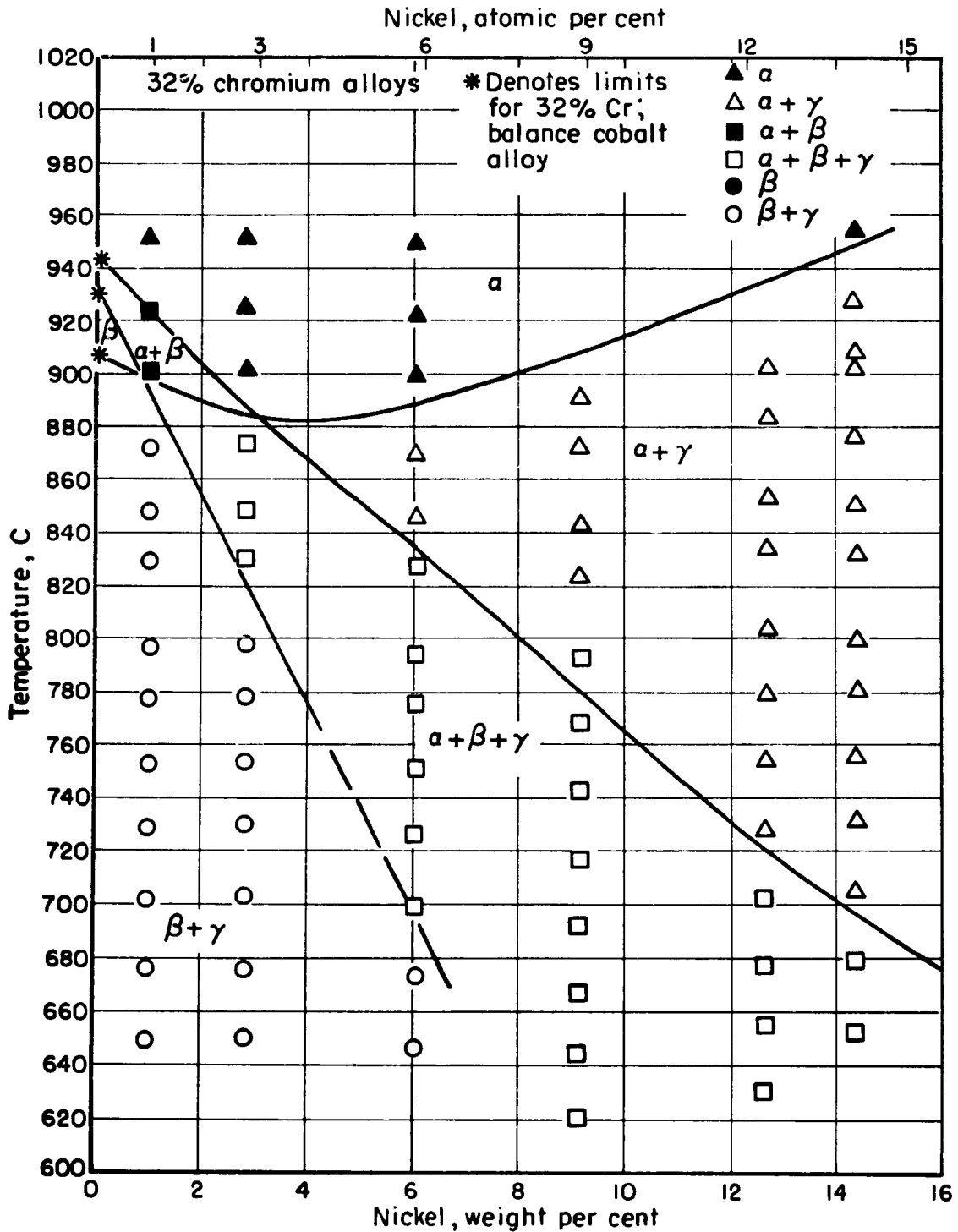


FIGURE 7. ALPHA-PLUS-BETA TEMPERATURE RANGE AND GAMMA PRECIPITATION AS A FUNCTION OF NICKEL CONTENT FOR COBALT-CHROMIUM-NICKEL ALLOYS CONTAINING 32 PER CENT CHROMIUM

A-1944

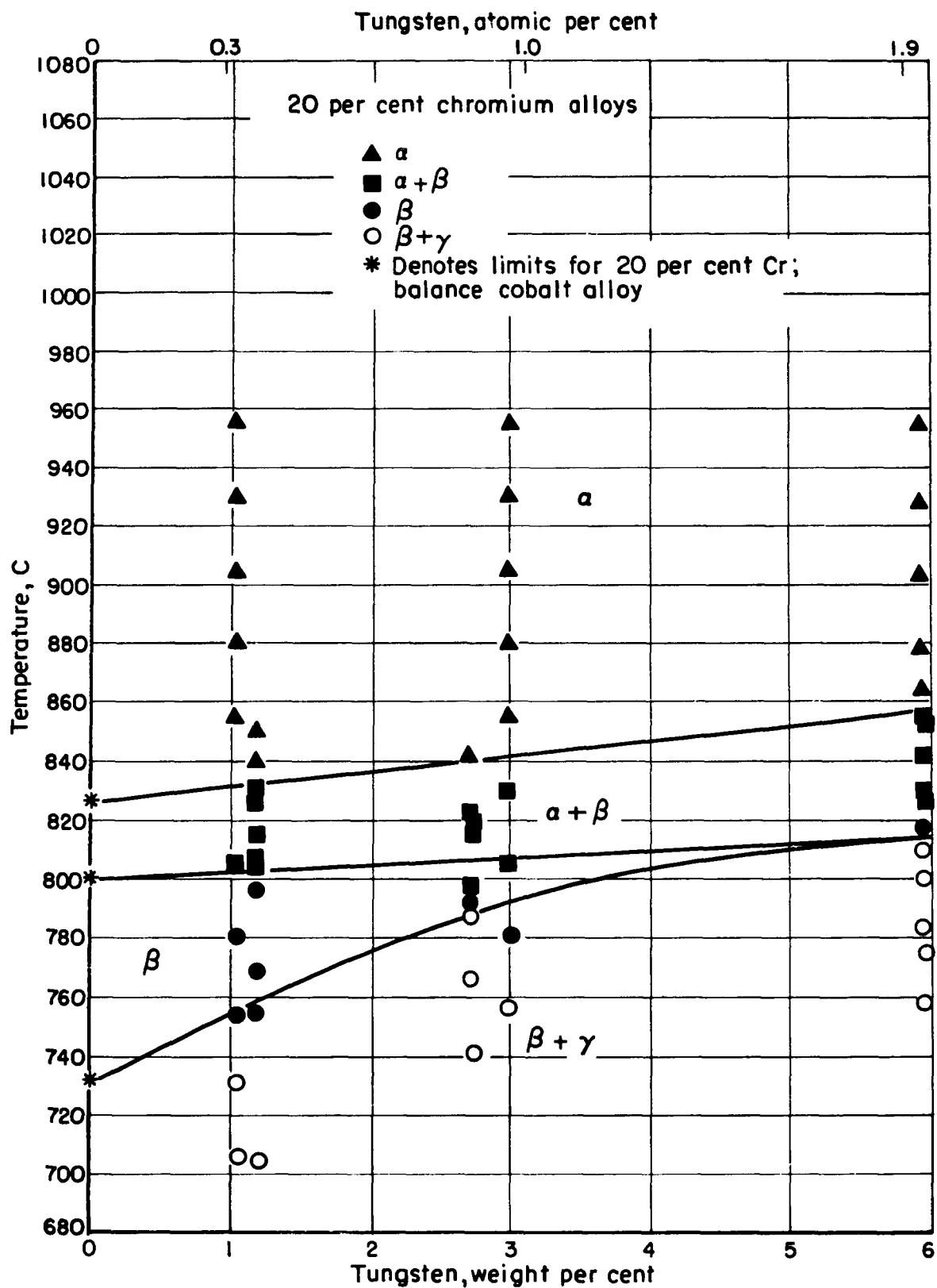


FIGURE 8. EFFECT OF TUNGSTEN ON ALPHA-PLUS-BETA TEMPERATURE RANGE AND GAMMA PRECIPITATION IN Co-Cr-W ALLOYS WITH  $\frac{\text{Cr} \times 100}{\text{Co} + \text{Cr}} = 20.0$

A-1945



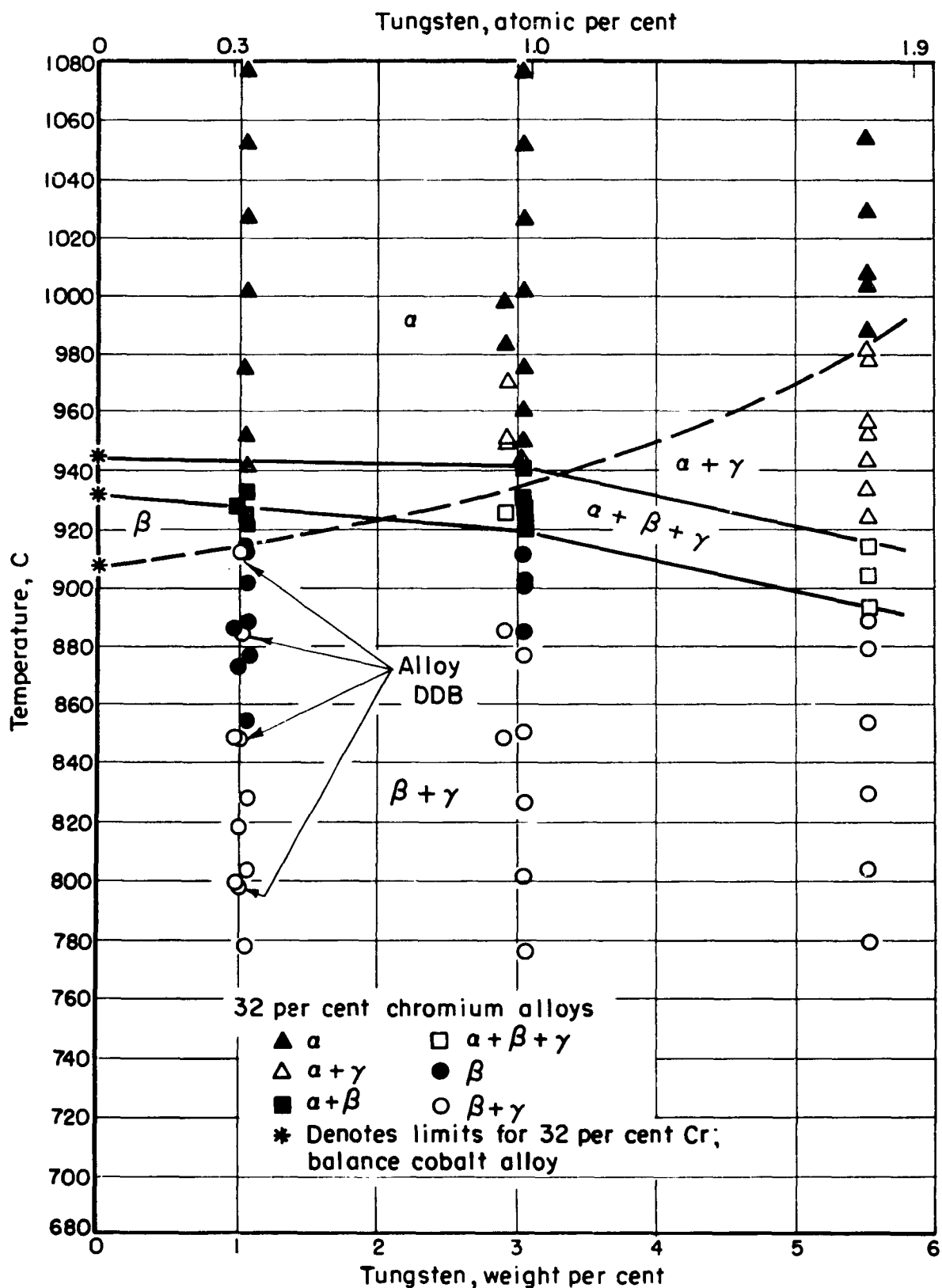


FIGURE 9. EFFECT OF TUNGSTEN ON ALPHA-PLUS-BETA TEMPERATURE RANGE AND GAMMA PRECIPITATION IN Co-Cr-W ALLOYS WITH  $\frac{\text{Cr} \times 100}{\text{Co} + \text{Cr}} = 32.0$

A-1946

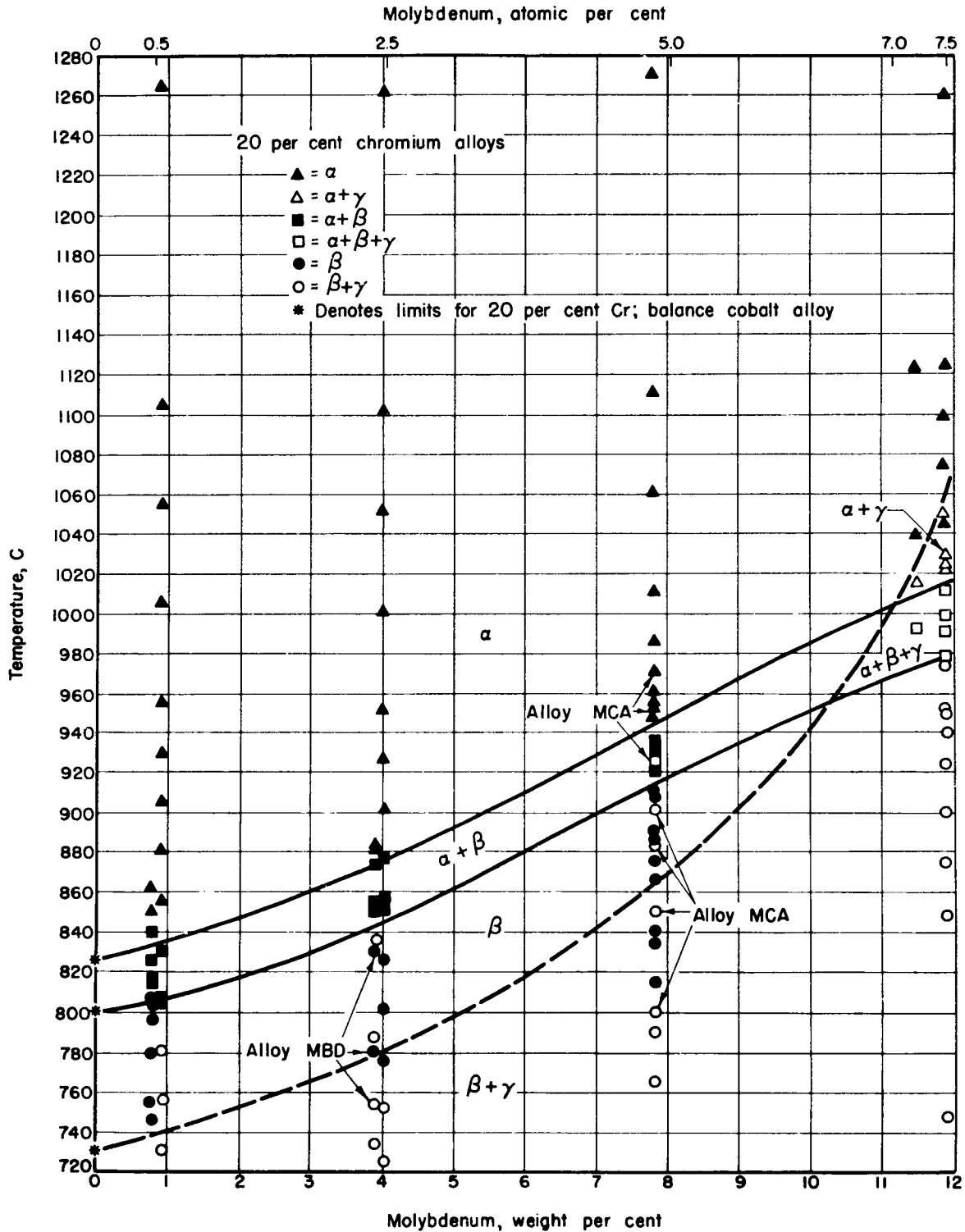


FIGURE 10. EFFECT OF MOLYBDENUM ON ALPHA-PLUS-BETA TEMPERATURE RANGE AND GAMMA PRECIPITATION IN Co-Cr-Mo ALLOYS WITH  $\frac{\text{Cr} \times 100}{\text{Co} + \text{Cr}} = 20.0$

C-1947

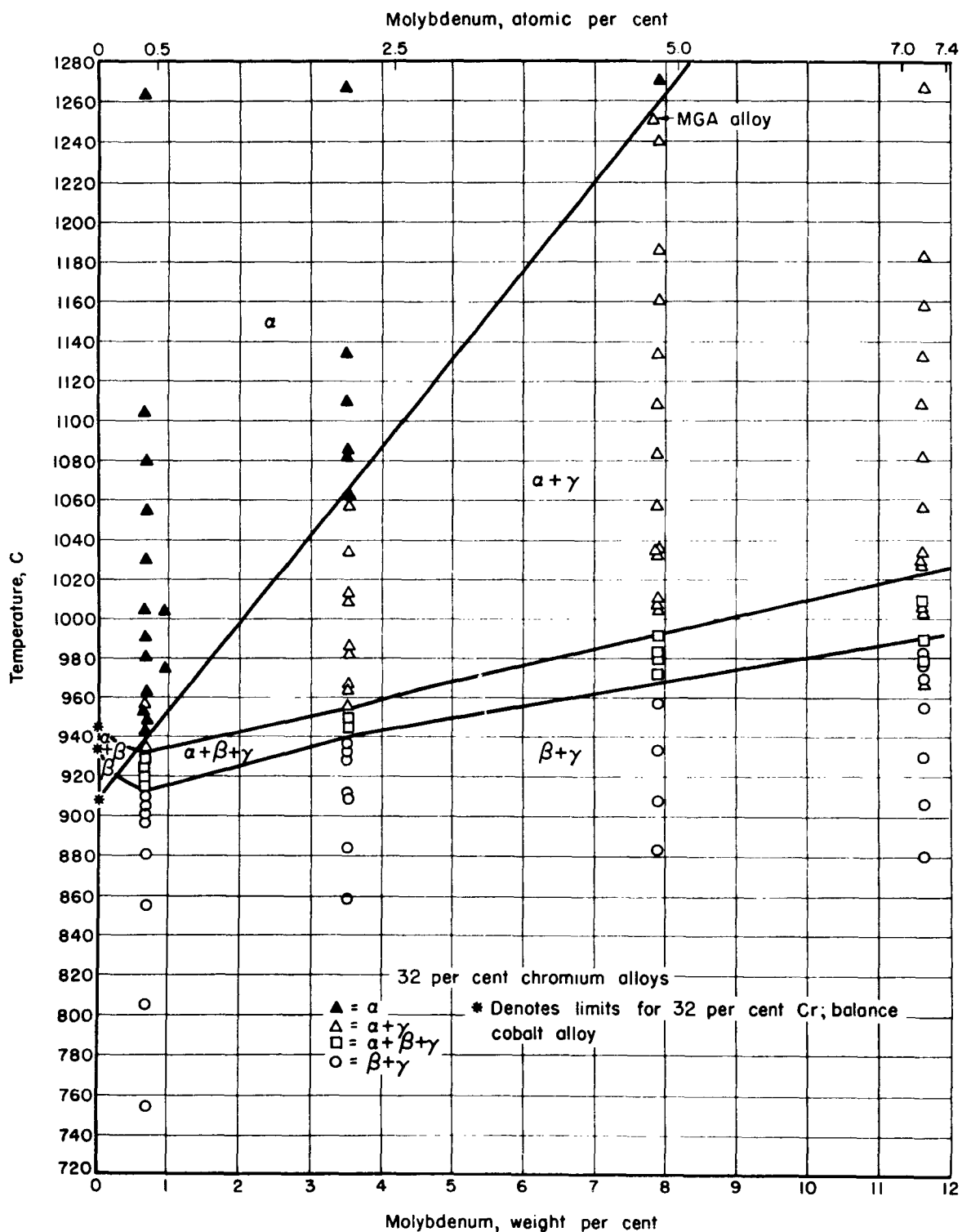


FIGURE II. EFFECT OF MOLYBDENUM ON ALPHA-PLUS-BETA TEMPERATURE RANGE AND GAMMA PRECIPITATION IN Co-Cr-Mo ALLOYS WITH  $\frac{\text{Cr} \times 100}{\text{Co} + \text{Cr}} = 32.0$

C-1948

further studies of this type would be discontinued and attempts would be made to study the phase relationships and properties of Vitallium (Stellite No. 21).

Study of Phase Relationships in  
Vitallium (Stellite No. 21)

Samples of Stellite No. 21 were subjected to X-ray diffraction and metallographic analyses to determine the temperature range for the alpha-beta transformation and to establish the identity and solution temperature for the various compound phases present. The following three alloys were prepared by the Haynes Stellite Division of the Union Carbide and Carbon Company for use in this investigation: the standard Stellite No. 21 alloy as prepared for commercial applications using part scrap; a similar alloy made from all raw melting stock; and a low-carbon modification of Stellite No. 21. The analyses of these alloys are contained in the Summary Report dated March 31, 1952.

Part of the stock from each of the three alloys was homogenized to eliminate alloy segregation. The homogenizing treatment used for these materials and the chemical analyses of the alloys in the as-cast condition and after homogenizing are presented in the Summary Report dated March 31, 1952. Specimens of the three alloys in both the as-cast and the as-homogenized conditions were aged at various temperatures for 100 hours; after these treatments, the phases present were identified by metallographic and X-ray diffraction methods.

Neither the metallographic etchants used on the binary and ternary cobalt-base alloys nor any of the etchants tried were successful in distinguishing between alpha and beta phases in the Stellite No. 21-type alloys. Consequently, X-ray diffraction analysis was used to identify the matrix phases present in the aged specimens.

Both metallographic and X-ray diffraction analyses were used to identify the minor or compound phases present in the specimens. Although several standard metallographic etchants were tried, heat tinting was found to be the most reliable means of differentiating these phases\*. The minor phases found in these specimens were gamma phase ( $\text{Co}_2\text{Cr}_3$ ),  $\text{Cr}_7\text{C}_3$ ,  $\text{M}_6\text{C}$ , and  $\text{M}_{23}\text{C}_6$ , which are described below:

Gamma phase is a cobalt-chromium intermetallic compound and it may contain small amounts of molybdenum or tungsten in solid solution. Gamma phase is darkened by heat tinting.

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\* This technique is described in the Summary Report dated March 31, 1952.

M<sub>23</sub>C<sub>6</sub> is essentially a chromium carbide with varying amounts of molybdenum, tungsten, and cobalt in solid solution. Upon heat tinting this carbide remains white.

M<sub>6</sub>C is a complex double carbide believed to consist of one atom of carbon, three atoms of noncarbide-forming elements, such as nickel or cobalt, and three atoms of carbide-forming elements, such as chromium, molybdenum, or tungsten. M<sub>6</sub>C is darkened by heat tinting.

Cr<sub>7</sub>C<sub>3</sub> is a simple chromium carbide which has been detected by the X-ray diffraction analyses, but has not been identified by metallographic methods.

Structural analysis of the aged specimens showed that the upper limit of the alpha-beta transformation temperature range in Stellite No. 21 was about 1000°C. Increasing the nickel content lowered this upper limit considerably, while carbon or molybdenum additions were quite effective in raising it. The effect of carbon was more pronounced in the homogenized material than in the as-cast material. Only the upper limit of the alpha-beta transformation temperature range could be determined, because of the sluggish nature of the reaction at low temperatures. However, there were some indications that the lower limit was approximately 700°C.

It is interesting to note that turbine blades in jet engines may operate frequently at temperatures where the two-phase structure is present.

In addition to the matrix phase or phases, the as-cast material contained patches of compound material in the interdendritic areas. These patches contained M<sub>23</sub>C<sub>6</sub> and M<sub>6</sub>C. Upon aging at low temperatures, a very fine precipitate formed in the vicinity of the interdendritic patches. As the aging temperature was increased, the precipitate became coarser and the M<sub>6</sub>C in the interdendritic patches started to go into solution. At a temperature of about 1100°C. the precipitate no longer formed, and at a temperature of about 1150°C. the M<sub>6</sub>C-type carbide all was taken into solution. The M<sub>23</sub>C<sub>6</sub>-type carbide persisted in these specimens to considerably higher temperatures.

The structure of the homogenized and quenched alloys consisted of a matrix phase (probably alpha) plus particles of M<sub>23</sub>C<sub>6</sub>-type carbides in the alloys of standard analysis; the low-carbon modification of Stellite No. 21 consisted of a single solid solution in the homogenized and quenched condition. Upon aging at low temperatures, a fine precipitate formed throughout the matrix phase of these alloys, generally on preferred planes. As the aging temperature was increased the precipitated particles became coarser, the maximum size being attained at a temperature of about 1000 to 1050°C.; as the aging temperature was increased further, less of the precipitate formed. The results of X-ray diffraction and metallographic analyses of these specimens showed that the precipitate consisted of gamma phase, M<sub>23</sub>C<sub>6</sub>, and M<sub>6</sub>C. However, above about 1100°C. only the M<sub>23</sub>C<sub>6</sub> carbide was formed.

## Mechanical Properties of Cobalt-Chromium-Base Alloys

### Creep-Rupture Tests on Binary and Ternary Alloys

Creep-rupture tests were made on cobalt-chromium binary alloys containing 20 and 32 per cent chromium, and on similar alloys containing nitrogen. These tests were conducted at temperatures of 780, 915, and 1000°C.

Some of these alloys had lower creep rates at higher stresses than at lower stresses, in tests conducted at the same temperature. Metallographic examination of the bars after testing showed that at higher stresses more gamma particles of a smaller size precipitated and effectively strengthened the alloys. Also, the test results indicated that the degree of prestress at the test temperature influenced the subsequent creep rate of these alloys. At a given test temperature, the higher the prestress, the lower was the creep rate. This was true even at temperatures above that at which gamma started to precipitate in alpha. It is possible that the precipitation of sub-microscopic particles was influenced by the degree of prestress and consequently produced this effect.

First-stage creep seldom was observed in the time-deformation curves for these specimens. In fact, there was usually a dip in the curve prior to the onset of second-stage creep and in some instances actual contraction occurred. A thorough analysis of this behavior led to the conclusion that time-deformation curves for these test specimens represented summations of two opposing reactions—one was the normal tendency toward creep, while the other was a contraction which may have resulted from the formation of submicroscopic particles of a second phase. Throughout this investigation the minimum creep rate was measured after the time-deformation-curve dip resulting from this contraction. This contraction phenomenon and the method of determining the minimum creep rate are described in detail in the Summary Report dated March 31, 1952.

The minimum creep rates obtained from the time-deformation curves were plotted as a function of stress for each of the alloys at each test temperature. From these curves, the stress to produce minimum creep rates of 0.0001 and 0.001 per cent per hour were determined; these values are plotted as a function of test temperature in Figure 12.

The curves in Figure 12 show that increasing the chromium or nitrogen content of the alloys increased their strength at all temperatures tested, except when the change in composition resulted in a phase change. These curves also show that alpha and beta had about the same strength. For example, in Figure 12a, the curve for the 20 per cent chromium binary alloy was approximately a straight line. At the two higher testing temperatures this alloy was alpha phase, whereas at the lower testing temperature it was beta phase. If there were an appreciable difference between the properties

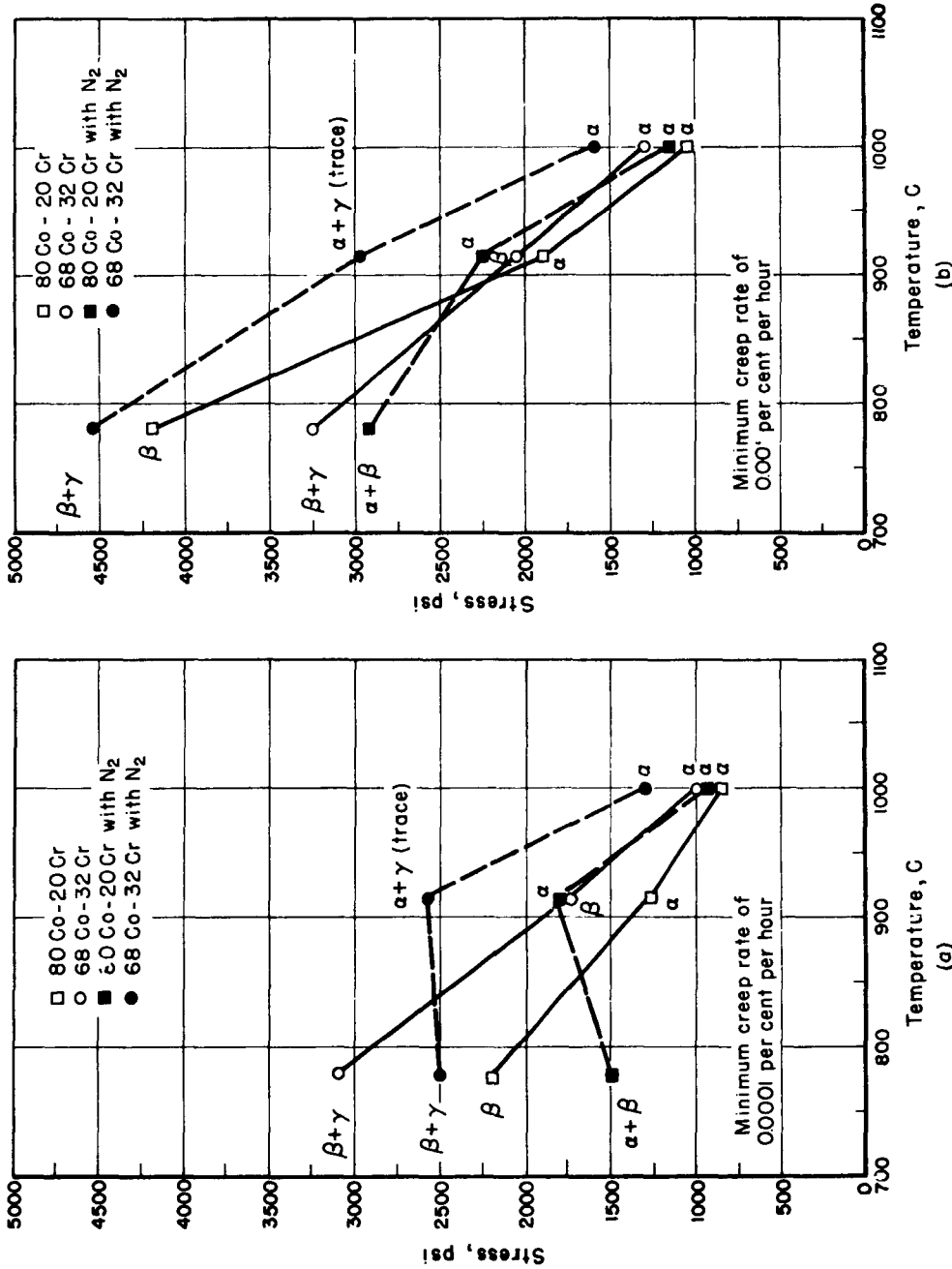


FIGURE 12. STRESS FOR A GIVEN MINIMUM CREEP RATE FOR COBALT-CHROMIUM BINARY ALLOYS AND COBALT-CHROMIUM-NITROGEN TERNARY ALLOYS CONTAINING 20 AND 32 PER CENT CHROMIUM. STRUCTURES SHOWN AT THE VARIOUS POSITIONS ARE THOSE INDICATED BY THE PHASE DIAGRAMS AND NOT THOSE ACTUALLY OBSERVED IN THE SPECIMENS

of alpha and of beta, the curve for this alloy probably would have deviated considerably from a straight line. Also, in this same figure, the 32 per cent chromium binary alloy and the 20 per cent chromium nitrogen-bearing alloy had about the same properties at the two highest test temperatures, respectively. At 1000°C. both alloys were alpha phase, while at 915°C. the 32 per cent chromium binary alloy was beta, whereas the nitrogen-bearing alloy was alpha. Furthermore, these curves show that a mixed alpha-plus-beta structure was weaker than either alpha or beta, under a given set of testing conditions. The 20 per cent chromium nitrogen-bearing alloy in Figure 12a was weaker at 780°C. than at 915°C.; at the lower temperature this alloy had a mixed alpha-beta structure, while at 915°C. it was all alpha. The curve for the 32 per cent chromium alloy containing nitrogen in Figure 12a shows the effect of gamma precipitation on these alloys. At the highest testing temperature, the alloy was all alpha with no gamma precipitate. At 915°C. a small amount of fine gamma precipitate was present and this apparently strengthened the alloy. At the lowest testing temperature the alloy was beta phase with a large amount of very coarse gamma precipitate. Here, again, the alloy was weaker at 780°C. than at 915°C. The 32 per cent chromium binary alloy also contained gamma precipitate in the matrix of beta phase at the lowest testing temperature; however, in this case considerably less gamma phase was present and the particles were relatively fine.

Further details regarding the testing methods and the results obtained are contained in the Summary Report dated March 31, 1952.

### Properties of Stellite No. 21-Type Alloys

Room-Temperature Hardness. Rockwell "C" hardness tests were made on aged specimens of Stellite No. 21-type alloys. These specimens represented the three modifications of Stellite No. 21 which were used in the structural study of this material, and had been aged both from the cast and from the homogenized and cold-worked conditions. The aging treatments consisted of holding at temperatures ranging from 500 to 1200°C. for 100 hours, followed by water quenching. Hardness versus aging temperature curves plotted from these data were presented in the Summary Report dated March 31, 1952.

The hardness versus aging temperature curve for each of the alloys investigated followed a characteristic pattern. In general, the as-cast stock began to age harden at about 650°C., reaching a maximum hardness of Rockwell "C" 32 to 40, depending upon the alloy, when aged at 700 to 750°C. At progressively higher aging temperatures, the hardness decreased slightly, up to about 1000°C., and then decreased more rapidly at higher temperatures. The homogenized alloys exhibited high hardness at the lowest aging temperatures because they had been cold worked prior to aging. At progressively higher aging temperatures, the homogenized and cold-worked material increased in hardness and reached a maximum at about 650 to 700°C.; this



hardness varied from Rockwell "C" 43 to 48, depending upon the alloy. The hardness of the homogenized and cold-worked material decreased more rapidly from this maximum hardness with increasing temperature than did that of the cast and aged material; when aged at approximately 1050°C., both the as-cast and the homogenized materials had about the same hardness.

The results of this study showed that variations in carbon, nickel, or molybdenum content of Stellite No. 21 had little effect on the aging characteristics of this alloy. Homogenizing and cold working resulted in a higher hardness after aging at all temperatures below 1000°C. and also slightly lowered the aging temperature at which the maximum hardness was attained.

Creep-Rupture Data. Creep-rupture tests were conducted to determine the 100-hour rupture strength at 1500°F.(816°C.) of Stellite No. 21 and the low-carbon modification of this alloy, in several conditions of heat treatment. The two alloys were tested as cast, as homogenized, and after several aging treatments. The aging temperatures selected were 725 and 975°C. These temperatures were chosen because the room-temperature hardness tests showed that aging these alloys at 725°C. resulted in maximum age hardening, while aging at 975°C. resulted in slight overaging. However, 975°C. is below the temperature at which the hardness decreases rapidly with increasing temperature. The Summary Report dated March 31, 1952, contains a detailed description of how these tests were conducted and the results obtained. Table 1 summarizes the results.

These data show that the as-cast material was considerably stronger than the homogenized or homogenized and aged material of the same alloy. The as-cast material was strengthened still further by aging at 975°C. In tests on the homogenized material, the specimens aged at 725°C. were weaker than those in any of the other conditions tested, even though this aging treatment resulted in the highest hardness at room temperature. It is shown further in Table 1 that Stellite No. 21 is considerably stronger than its low-carbon modification under all test conditions; this indicates that an increase in carbon content strengthens this alloy.

Hot-Hardness Data. In order to determine whether the hot-hardness test might be used to predict the high-temperature strength of cobalt-chromium-base alloys, hot-hardness tests were made at 1500°F.(816°C.) on the same alloys in the same conditions of heat treatment as were used for the 100-hour rupture tests. These hardness tests were not made on the as-cast or as-cast and aged material because of the heterogeneity of the structure. The tests were made in a vacuum chamber heated to 1500°F.(816°C.) using a standard Vickers-type diamond indenter and a load of 1/2 kg. applied for 15 seconds. The results of these tests also are shown in Table 1. No direct relationship between the 100-hour rupture strength and the hot hardness of these alloys is apparent.

TABLE 1. STRESS TO PRODUCE RUPTURE IN 100 HOURS AT 1500°F. (816°C.)  
COMPARED WITH HARDNESS AT 1500°F. FOR TWO HEATS OF  
MODIFIED STELLITE NO. 21 ALLOY

Alloy	Initial Condition of Alloy	Stress to Produce Rupture in 100 Hours, p.s.i.	Hardness at 1500°F., Vickers DPHN
Low-carbon (0.14% C) modification of Stellite No. 21 made from all raw melting stock	As cast	18,200	---
	Cast and aged at 975°C.	19,200	---
	As homogenized	14,500	145
	Homogenized and aged at 725°C.	13,800	188
	" " " " 975°C.	16,100	158
Modified Stellite No. 21 of standard analysis, but made from all raw melting stock (0.32% C)	As cast	23,400	---
	As homogenized	21,500	172
	Homogenized and aged at 725°C.	18,200	220
	" " " " 975°C.	20,300	192

Vickers hardness tests were made on these specimens at room temperature also. It was found that the hardness at 1500°F. was approximately one-half the room-temperature hardness for each of the specimens.

May 29, 1952

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## APPENDIX

## APPENDIX

### List of Technical Reports

1. Summary Report dated November, 1947
2. Summary Report dated September 20, 1949
3. Summary Report dated December 15, 1950
4. Summary Report dated March 31, 1952

### List of Publications

1. Elsea, A. R., A. B. Westerman, and G. K. Manning, "The Cobalt-Chromium Binary System", Metals Technology, June, 1948 (Technical Publication No. 2393); also, Trans. AIME, Vol 180, 1949, pp 579-602.
2. Elsea, A. R., and C. C. McBride, "The Effects of Nitrogen, Iron, or Nickel Upon the Alpha-Beta Transformation and Gamma Precipitation in Cobalt-Chromium Alloys", Journal of Metals, Vol 188, No. 1, January, 1950, pp 154-161; also, Trans. AIME, Vol 188, pp 154-161.
3. Fletcher, E. E., and A. R. Elsea, "Effects of Tungsten or Molybdenum Upon the Alpha-Beta Transformation and Gamma Precipitation in Cobalt-Chromium Alloys", Journal of Metals, Vol 3, No. 10, October, 1951, pp 897-902; also, Trans. AIME, Vol 191, 1951, pp 897-902.

May 29, 1952

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